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STRUCTURE AND PROPERTIES OF GLASSES.(U)
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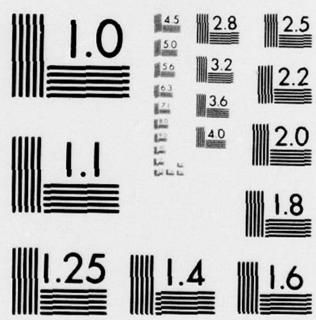

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UNIVERSITY OF CALIFORNIA, LOS ANGELES

LOS ANGELES, CALIFORNIA

FINAL SCIENTIFIC REPORT

to

AIR FORCE OFFICE OF SCIENTIFIC RESEARCH

on project entitled

"STRUCTURE AND PROPERTIES OF GLASSES"

See 1473

Grant No.: AFOSR 75-2764

Inclusive Dates: September 1, 1974 to September 30, 1978

Principal Investigator: Dr. John D. Mackenzie
Professor of Engineering and Applied Science

November, 1979

1. Introduction

Because of its transparency, ease of fabrication, superior chemical durability and desirable mechanical properties, glass has long been an important material of engineering. Despite the long history of glass technology, glass science is actually a fairly recent development. The relatively small amount of scientific research on glasses up to the present has already resulted in significant improvements of the properties of old glasses and the development of new glasses. However, our knowledge is still insufficient to permit exact explanation or predictions or properties from chemical compositions. This is mainly because of the lack of structural understanding of glasses.

The broad objective of this research is a better understanding of the relation between structure and properties of inorganic glasses. From such structural knowledge, glasses of controllable and predictable properties can be prepared by controlling their chemical compositions. In addition, when the glass-forming regions of multi-component phase diagrams are known, it is possible to predict the ultimate limits of certain properties, for instance, strength and elastic modulus.

2. Summary of Scientific Background on this Project

This application of structural understanding to the preparation of glasses of superior properties has already been demonstrated for some oxide glasses under a previous research grant supported by AFOSR at the University of California at Los Angeles. The results of that research program became the basis of the present program. It is thus desirable to briefly review the highlights of the previous project.

a) Glasses of Superior Hardness

From work on flow under pressure, we had achieved a significant degree of understanding of the hardness of glass. A relation was developed for the direct calculation of hardness of glass.

For over 100 different silicate glasses, calculated and observed hardness values were found to be in agreement to $\pm$ 10%.

We then proceeded to utilize this equation to predict hardness from chemical compositions. A large number of new glasses were melted and their hardnesses measured. Prior to our work, the hardest glass known was silica glass, the Vickers hardness of which is 540 kg/mm^2. The hardest glass we prepared had a Vickers value of 1026 kg/mm^2, some 60% harder.

b) Direct Calculation of Young's Modulus

An equation was derived for the direct calculation of Young's Modulus of a wide variety of glasses from chemical compositions.[1] Excellent agreements were obtained for silicate glasses and borate glasses. This development now permits the prediction of what glass compositions will have the highest modulus and hence can lead to the preparation of glasses of the highest modulus possible. This is important not only for bulk glass such as windows but also for glass-fibers which are being used in composite systems.

c) Ion Exchange Under Pressure

The ion-exchange process of strengthening glass consists of the immersion of a solid piece of glass in fused salts.[2] The smaller ions in the glass, such as Li^+ and Na^+ ions will exchange with larger ions in the fused salt such as K^+ ions. The glass is kept in the constant volume or solid state, i.e., under T_g so that the ion-exchange will result in a surface layer of high compression. The strengths of oxide glasses have been increased from approximately 10,000 psi to 100,000 psi by this method.

From the results of our studies on ionic conductivity and viscous flow under pressure, we considered that studies of ion-exchange under pressure was highly desirable. Preliminary calculations indicated that if the pressure was increased to 1 kilobar, the temperature could be raised about 70° and the glass

would still remain rigid. The electrical resistivity would have dropped by about an order of magnitude this way. The above ideas were based on the assumption that ion-exchange was directly prportional to ionic conductivity. If this approach was correct, the ion-exchange process could be accelerated very significantly.

A high pressure was thus designed and constructed. Ion-exchange experiments were carried out successfully under elevated pressures. The preliminary results obtained were encouraging. This approach opened up significant possibilities in obtaining ultra-high strength glasses with a new process which could be economically exploited.

The results obtained were not only promising and interesting but also furnished us with new structural understanding of oxide glasses. It seemed likely that additional research utilizing the same approach would be desirable. The present research project was then initiated. This is described below.

3. Research Carried Out

The currently concluded research program under Grant No. AFOSR 75-2764 was first proposed in 1974 and the program was reviewed and approved at yearly intervals. The various research activities carried out from 1974 to 1978 are briefly described below. They can be conveniently divided into two groups.

a) Thermal Expansion of Oxide Glasses

The applications of oxide glasses are frequently limited by their expansion coefficients. For instance, an oxide glass with a high expansion coefficient is susceptible to thermal shock. An oxide glass with a low expansion coefficient cannot be easily laminated to organic plastics. Because glasses of both high and low expansion coefficients are of importance to the Air Force, our research was directed towards a total understanding of the phenomenon of thermal expansion.

b) Ion-Exchange of Oxide Glasses

Ion-exchange can be utilized to strengthen glasses appreciably. It can also be utilized to modify the surface property of glasses such as chemical durability and refractive index. Because both strength and optical properties of glasses are of importance to the Air Force, our research could affect these properties.

Important research accomplishments are summarized below:

4. Research Accomplishments

a) Thermal Expansion of Oxide Glasses

When an oxide glass is laminated to an organic plastic, the stress developed at the interface is given by the following equation:

$$S = (T_1 - T_2)\,(\alpha_P - \alpha_G)\,\frac{E_P\,E_P}{E_P + E_G}$$

T_1 = temperature of lamination

T_2 = lowest temperature of use of window

α_P, α_G = expansion coefficients of plastic and glass respectively

E_P, E_G = Young's modulus of plastics and glass respectively

If we assume T_1 to be 250°C and T_2 to be -50°C, we can calculate S, the stress at the interface for two cases.

Case I -- Plastic used is Polyvinyl Chloride

$\alpha_P = 550 \times 10^{-7}$ per °C

$E_P = 0.37 \times 10^{6}$ psi

(a) For Window Glass

$\alpha_G = 85 \times 10^{-7}$ per °C

$E_G = 9 \times 10^{6}$ psi

$S_{calculated}$ = 5139 psi

b) For <u>High Expansion Glass</u>

$\alpha_G = 250 \times 10^{-7}$ per ^oC

$E_G = 6 \times 10^6$ psi

$S_{calculated}$ = <u>3110 psi</u>

<u>Conclusion</u>: The decrease in the stresses generated can be as much as 60%! This is true even if the real values of the stresses developed are lower. If we can develop a glass of higher α, the decrease in stress can be lower than 50%.

<u>Case II</u> -- Plastic used as Polyvinyl Butyral

$\alpha_P = 154 \times 10^{-7}$ per ^oC

$E_P = 3.7 \times 10^6$ psi.

a) For <u>Window Glass</u>

$\alpha_G = 85 \times 10^{-7}$ per ^oC

$E_G = 9 \times 10^6$ psi

$S_{calculated}$ = <u>5460 psi</u>

b) For <u>High Expansion Glass</u>

$\alpha_G = 150 \times 10^{-7}$ per ^oC

$E_G = 7.7 \times 10^6$ psi

$S_{calculated}$ = <u>300 psi</u>

<u>Conclusion</u>: The stress developed can be practically <u>all</u> eliminated if high expansion glass is available.

It is thus easily seen that oxide glasses of high expansion coefficients are very desirable for laminated windows. A review of the literature indicated that the expansion of glasses was not well-understood and that oxide glasses of high expansion were not being offered by industry for windows. We therefore,

proposed to develop a theory for the calculation of thermal expansion of glasses from their chemical compositions. High expansion glasses would then be made.

Theoretical studies led to the development of an equation for the calculation of thermal expansion. The equation was:

$$\alpha = \frac{\rho\left\{11.2(V_t-0.4)\sum(R_c/R_a)X_i\right\}\left\{4.1\sum C_pX_i\right\}}{(3V_t^2\sum G_iX_i\ \sum M_iX_i)\times 10^4}$$

where ρ is density, V_t is packing density, R_c/R_a is ratio of cation to anion size, X_i is bond number fraction, C_p is heat capacity, G_i is dissociation energy per unit volume, M_i is molecular weight. Calculated expansion agrees well with observed expansion for some 150 glasses.

In an experimental study, over 50 phosphate glasses were prepared and their expansion coefficients and chemical durability studied. Very high expansion glasses (up to 286×10^{-7}) of relatively good chemical durability were successfully prepared.

Glasses of high expansion coefficient usually have poor chemical durability. A concept was developed to use a high expansion glass of poor chemical durability and to ion-exchange it so that the surface layer would be of good chemical durability. Since the bulk of the glass would control the overall expansion, the high expansion would be retained.

A number of $Na_2O.SiO_2$ glasses of high expansion and poor chemical durability were prepared and ion-exchanged in molten $LiNO_3$. The chemical durability has increased by some 3½ orders of magnitude. In addition, the Li_2O-rich surface produced a slight compressive effect and gave an average of 20% increase in the tensile strength. Samples of such glasses were laminated to organic polymers and showed great improvements over ordinary window glass in that delamination no longer occurred on cooling. Similar results were obtained with

phosphate glasses.

Because of the need of very low expansion glasses and glass-ceramics which would minimize thermal shock on exposure to lasers, we have been studying the system Cu_2O-Al_2O_3-SiO_2. Glasses from this system have unexpectedly low expansion.[3] The reasons were unknown. The copper ions exist as Cu^+ and Cu^{2+} in the glass. The ionic radii of these ions are practically the same as Na^+ and Zn^{2+}. We thus prepared Na_2O. ZnO. Al_2O_3. SiO_2 glasses. Their expansions were very high. The size and valence of the copper ions were therefore not responsible for low expansion. Electron microscopy revealed that the glasses were phase-separated. Presumably, a low expansion matrix phase controls the expansion. Although we were unsuccessful in the preparation of new, transparent, low expansion glasses, understanding of why the copper aluminosilicate glasses have such low expansions was obtained.

In the course of heat-treatment of the Cu_2O. Al_2O_3. SiO_2 glasses, a new family of glass ceramics was discovered which have negative, zero, and small positive expansions and high temperature stability, (to 1000°C). The additions of minor amounts of B_2O_3, ZnO, Na_2O, etc. lowered the melting temperatures of the glass but did not effect the expansion of the glass-ceramics.

b) Ion-exchange of Oxide Glasses

In the course of this research, it was discovered that if a glass were to be subjected to hydrostatic pressure for a short time at an elevated temperature, (300 atms. 2 hrs., 325°C, for instance) and then this glass was ion-exchanged in a fused salt bath at 1 atmosphere, very significant increased strengthening was possible. For example, without this pre-treatment, a glass with a tensile strength of 16,000 p.s.i. can be strengthened to 43,000 p.s.i. With the pre-treatment, the strength is increased to 64,000 p.s.i.

This very interesting behavior through the pretreatment process can have important practical consequences since very high strength windows can now be made easier and cheaper.

A variety of experimental tools were use in an attempt to reveal the exact nature of this pressure treatment. Electron-microprobe, Auger electron microscopy, ESCA and Secondary Ion Mass Spectrometer analysis were used. However, the results were ambiguous. They did, nevertheless, indicate that slight variations on the surface of the glass had occurred after the pressure treatments. Very careful weight change experiments were made after ion-exchange and the results of samples with and without the pressure treatment compared. Apparently, the ion-change after pressure treatment was more rapid. That such minor alterations on the glass surface could have such major effects on ion-exchange was unexpected and warranted further study.

Careful considerations of the ion-exchange process indicated that the elastic modulus of the glass must be affected and must also be considered before one could attempt to understand the strengthening process. Surprisingly, it was discovered that there was no published information on the effects of ion-exchange on elastic modulus. This work suggested that a study of the relation between ion-exchange and elastic modulus would be important and could lead to the preparation of oxide glasses of high elastic modulus.

5. Relevance of this Program to Air Force

Glass is an important structural, optical and electronic material to the Air Force. The general relevance of a study of the relationship between glass structure and glass properties to Air Force needs is obvious. More specifically, the important implications of the present research are:

a) Improved laminated windows for aircraft

b) Stronger, harder and cheaper strengthened glasses

c) Laser resistant glasses and glass systems for windows and missiles.

6. Other Achievements

a) Primarily because of continuing opportunity to conduct research on glass under AFOSR sponsorship, J.D. Mackenzie was elected to the National Academy of Engineering in 1966 for contributions to glass science and technology.

b) J.D. Mackenzie was invited by the Japan Ceramic Association to be its Distinguished Lecturer in 1976, being the first foreign scientist so honored.

c) J.D. Mackenzie has continued as Editor-in-Chief of the Journal of Non-Crystalline Solids which has now become the foremost international journal on glass science.

d) J.D. Mackenzie was awarded a visiting professorship by the Japan Association for the Promotion of Science in 1978 and lectured in Japan for six weeks on glass science.

e) The following thesis were awarded to the students named below, based on research supported by AFOSR:

J. Wakaki, M.S. J. Crandall, M.S.

K. Kadokura, M.S. R. Almeida, M.S.

7. Publications in this Period

"Development of Glasses for Some New Applications," Proc. Tenth Inter. Cong. on Glass, 4, 71-81 (1974), by J.D. Mackenzie

"Preparation of Low Dielectric Constant Glasses," J. Non-Crystalline Solids, 16, 313-314 (1974) by F. Chen, B. Dunn and J.D. Mackenzie

"Vickers's Hardness of Glass," by J. Non-Crystalline Solids, 15, 153-164 (1974) by M. Yamane and J.D. Mackenzie

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"Desalination Using Porous Glass Tube Coated with Thin Cellulose Acetate," Yogyo-Kyokai-Shi, 83, 507-510 (1975) by A. Makishima and J.D. Mackenzie

"Preparation of Hard Glass," submitted to AFOSR; approval to publish not yet granted because of patent possibility, by K. Park and J.D. Mackenzie

"Structure and Ionic Properties of Glass," Int. Rev. of Science, Phys. Chem. Series 2, 6, Ed. by J. Bockris, Butterworths, London (1975) by B. Dunn and J.D. Mackenzie

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"Transport Properties of Glass-Silicon Heterojunctions," J. App. Phys., 47, 1010-1014 (1976) by B. Dunn and J.D. Mackenzie

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"Recent Important Advances in the Science and Technology of Glass and Ceramics in the U.S.A.," Ceramics Japan, 12, 91-97 (1977), by J.D. Mackenzie

"High Temperature Electrolytes - The Past Thirty Years," Chap. 22 in book ELECTROCHEMISTRY: THE PAST THIRTY AND THE NEXT THIRTY YEARS, Edited by H. Bloom and F. Gutman, Plenum Publishing Corp. (1977), by J.D. Mackenzie

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"Nickel Sulfide Stones in Glass", Glass Ind., 59, 32-33, (1978) by J.D. Mackenzie

"New Applications in Glass," J. Non-Crystalline Solids, 26, 456-481 (1978) by J.D. Mackenzie

"Glasses and Glass-Ceramics from Naturally Occurring $CaO-MgO-Al_2O_3-SiO_2$ Materials. (I) Glass Formation and Properties," J. Non-Crystalline Solids, 27, 75-81 (1978), by L.J. Shelestak, B. Dunn and J.D. Mackenzie

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8. Personnel

Dr. J.D. Mackenzie, Principal Investigator

Dr. T. Minami, Research Assistant,	1974-75
Dr. K. Matusita, Research Assistant,	1975-77
Dr. R. Ota, Research Assistant,	1977-78
Mr. J. Wakaki, Research Assistant,	1974-78
Mr. J. Ericson, Research Assistant,	1974-75
Mr. C.M. Baldwin, Research Assistant,	1975-77
Mr. K. Kadokura, Research Assistant,	1977-78
Mr. J. Crandall, Research Assistant,	1976-77
Mr. R. Almeida, Research Assistant,	1977-78

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2. J.D. Mackenzie, Vistas in Science, p. 94-115, University of New Mexico Press (1968).

3. T. Baak, U.S. Patent 3,414,465 (1968).

COMPLETED PROJECT SUMMARY

1. TITLE: Structure and Properties of Glasses

2. PRINCIPAL INVESTIGATOR: Dr. John D. Mackenzie
Materials Department
University of California
Los Angeles, California 90024

3. INCLUSIVE DATES: 1 September 1974 - 30 September 1978

4. GRANT NO.: AFOSR-75-2764

5. COSTS AND FY SOURCES: $28,280 FY 75;
$44,840 FY 76;
$55,913 FY 77;
$55,805 FY 78.

6. SENIOR RESEARCH PERSONNEL: Dr. T. Minami, Dr. K. Matusita, Dr. R. Ota

7. JUNIOR RESEARCH PERSONNEL: J. Wakaki, J. Ericson, C.M. Baldwin, K. Kadokura, J. Crandall, R. Almeida

8. PUBLICATIONS:

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9. Abstract of Objectives and Accomplishments

Glasses are solidified liquids without long-range order. It is usually difficult to obtain structural information by direct tools such as X-ray diffraction. The relationship between properties and chemical composition is thus difficult to obtain in the absence of structural information. The objectives of this research are to obtain structural information and indirect information from the measured results of some properties. From such structural information and knowledge of chemical composition, it was planned that predictions can be made of properties of some oxide glasses.

In this project, theoretical considerations have led to the development of an equation which permitted the calculation of thermal expansion from a knowledge of chemical composition. Many new glasses of high expansion coefficients were subsequently prepared. The chemical durability of these glasses was significantly improved by ion-exchange of the glasses in fused salt. It was discovered that phase separation in some indicate glasses could result in practically zero expansion coefficients.

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REPORT DOCUMENTATION PAGE

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1. REPORT NUMBER: 18 AFOSR-TR-80-0052 — 2. GOVT ACCESSION NO	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle): 6 Structure and Properties of Glasses.	5. TYPE OF REPORT & PERIOD COVERED: Final Scientific 9-1-74 to 9-30-78
	6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s): 10 John D. Mackenzie	8. CONTRACT OR GRANT NUMBER(s): 15 AFOSR-75-2764
9. PERFORMING ORGANIZATION NAME AND ADDRESS: University of California, Los Angeles, Hilgard Avenue, Los Angeles, CA 90024	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS: 61102F 16 2303 A3 17 A3
11. CONTROLLING OFFICE NAME AND ADDRESS: Directorate of Chemical Sciences /NC, Air Force Office of Scientific Research, Bolling AFB, D.C. 20332	12. REPORT DATE: 11 Nov[redacted]79
	13. NUMBER OF PAGES: 16
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office): 12 17	15. SECURITY CLASS. (of this report): Unclassified
	15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

DDC RECEIVED JAN 30 1980 E

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

9 Final ~~scientific~~ rept. 1 Sep 74-30 Sep 78,

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Chemistry, Basic Research, Air Force Office of Scientific Research Glass, Structure, Strength of Solids, Expansion of Solids, Elastic Properties

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

A theory was developed to calculate the expansion coefficient of oxide glasses and new glasses of high expansion prepared phase separation was found to have large effects on expansion. When the surface of glasses was treated by pressure, the ion-exchange rate was significantly increased. The elastic properties were affected by ion-exchange.

072 250 gm